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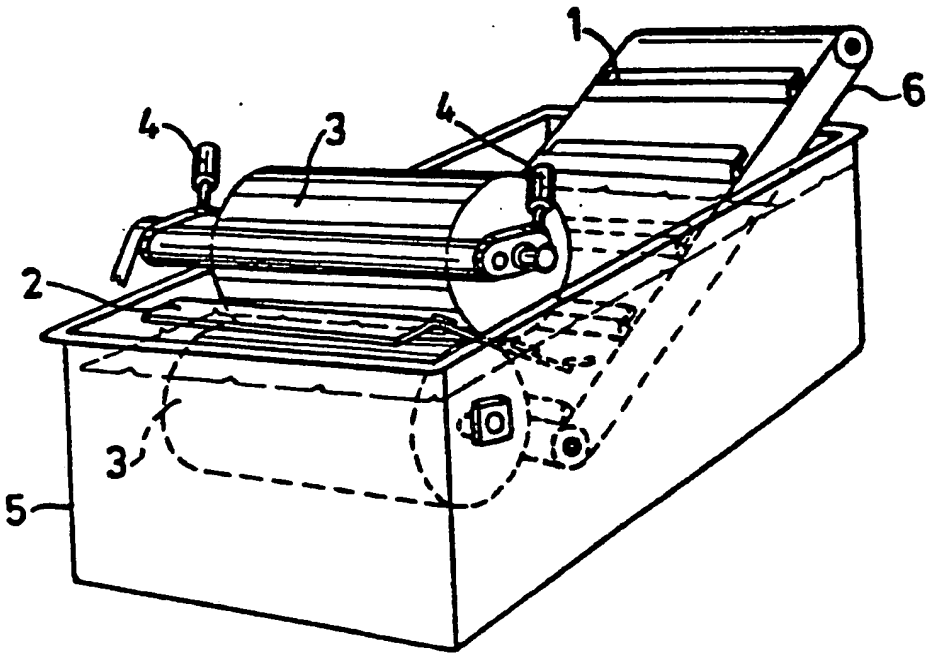
(54) Title: METHOD OF IMPREGNATING LIGNOCELLULOSE MATERIAL



(57) Abstract

Lignocellulose material in the form of pieces with a length in the fibre direction of at least 100 mm is compressed perpendicularly to the fibre direction of the material to such a degree, that its pores substantially are compressed. The material then is allowed to expand in impregnation liquid. Thereafter the material is disintegrated to fibre bundles and/or individual fibres.

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Method of impregnating lignocellulose material

This invention relates to a method of impregnating lignocellulose material in piece form with a length in fibre direction of at least 100 mm.

The lignocellulose material which, for example, can be different kinds of wood or bamboo, is intended for the manufacture of pulp to be used in different paper and cardboard qualities etc.

At all pulp production comprising treatment of the starting material with a liquid, which possibly includes chemicals, it is of greatest importance, that the liquid gets into contact with the entire material amount as intimately and uniformly as possible. This is normally achieved by chipping the material to chips, which then are soaked with the liquid. It is tried by different methods, such as vacuum evacuation, pre-steaming, hydrostatic pressure etc., to facilitate the penetration of the liquid into the chip pieces. The resistance of the fibre material to liquid penetration, the irregular form of the chip pieces etc., however, render it difficult to achieve rapid and uniform impregnation of the chips.

The complex of impregnation problems is intimately associated with the structure of the fibre material. In the following the structure of wood is dealt with, but the reasoning can be applied to other lignocellulose materials.

The solid wood substance has for all kinds of wood a density of about 1,50. The wood further includes a varying amount of pores in the form of lumens, i.e. the central cavity of the fibres and other wood cells, and other cavities, which are filled with air or liquid, so that the density of the dry wood varies between about 0,2 for balsa and 1,2 for guajacholz.

The wood is hygroscopic and takes up water by chemical sorption, adsorption in the cell walls and capillary condensation in the submicroscopic structure of the wood substance. This water take-up, which takes place causing swelling, continues up to the so-called fibre saturation point, at which the submicroscopic capillaries are filled with water. The moisture quotient of the wood, i.e. the moisture content based on bone dry wood, then is in an average 28% and varies between 22% for coniferous kind of wood, which is heavily resinous, and 32-35% for certain deciduous kind of wood, a.o. beech, according to Kollmann, Technologie des Holzes und der Holzwerkstoffe, second edition, first volume, Springer Verlag 1951, page 394. Above the fibre saturation point, the wood then can take up additional water by filling lumens and other cavities in the wood. This take-up takes place without increase in volume and depends on the porosity of the wood and can amout to 200-300% or more for highly porous kinds of wood. According to Kollmann, page 333, the following applies to wood, which has taken up water to the fibre saturation point or above said point

$$R_{max} = \frac{1,50}{1+0,28 \cdot 1,50} = 1,056 \text{ kg/dm}^3$$

where R_{max} is the maximum dry density of the wood free of pores at swelling maximum.

At impregnation it is desired to fill the pores of the wood with impregnation liquid as completely as possible. Then the delignification at the reaction of the wood with the chemicals is rapid and uniform, so that the reaction time can be kept short and the pulp produced has a high and uniform quality.

The impregnation can be carried out in different ways, for example by atmospheric suction or by pressure impregnation, at which the liquid is pressed into the wood by means of hydrostatic pressure. These methods

and similar ones have the disadvantage, that air remaining in the pores is locked up and prevents the liquid to enter. It is, therefore, tried in different ways to drive the air out, by steaming, vacuum suction etc.

It is usual to impregnate wood in chip form by compressing it and cause it to expand in the liquid. In most cases this is carried out by compressing the chips in a compressing screw conveyor, for example according to SE-PS 160636. The application of the pressure, however, which is required for effectively compressing the pores of the wood results in so strong friction forces and so great wear of the apparatus equipment, that it is practically unfeasible. The compression in a screw feeder for chips, therefore, in practice consists to the greatest extent of compacting the chips, so that air and possibly water between the chip pieces are driven out.

In order to achieve higher compression, it was also tried to press chips between rolls, for example according to CA-PS 677 418. Due to small particles of non--uniform size and shape constituting the chips, however, this method is difficult and therefore not applied in practice.

Mechanical pulps are manufactured of wood in two ways, which are different in principle, viz. by grinding blocks or refining chips. At grinding, the block, i.e. the cross-cut logs, is pressed against a rotating grindstone while water is being added. At refining, the chips are disintegrated to pulp between two refining discs rotating relative to each other. The method can be completed by heating the chips with steam at overpressure prior to and during the refining, so-called thermomechanical process, and/or impregnating and possibly digesting the chips with a chemical solution prior to the refining, so-called chemi-mechanical process.

By these methods, especially the chemi-mechanical one, long-fibred pulps are obtained, the strength properties of which are better than those obtained at grinding, but the optical properties are somewhat worse. The great disadvantage, however, is the very high energy consumption, which is substantially higher than at grinding. This is thought to be due to the fact, that the chip pieces whirl about between the refining discs without definite orientation of the fibres, while at grinding the block is held steady and is pressed against the grindstone with the fibres oriented in one and the same direction, i.e. in the plane of the grinding surface perpendicularly to its direction of movement. Only the outermost thin wood layer closest to the grinding surface is defibered, while the remaining part of the wood is unaffected and does not consume any unnecessary energy.

In order to make the groundwood pulp more long-fibred and stronger, the block was impregnated and digested, for example according to US-PS 2 713 540. This method, however, is very troublesome and results in an unimpregnated core, which assumes dark colour at digestion.

Impregnated chips also have been ground, for example according to Tappi Journal, May 1987, vol. 70, No. 5, page 119-123. As the chip pieces are oriented random in relation to the grinding surface and its direction of movement, the resulting pulp is non-uniform and has high shives content.

It was found practically impossible to orient the chips at grinding, due to the shape of the chip pieces and their short extension in the fibre direction.

In the patent application SE 8604769-3 a method has been proposed to manufacture mechanical pulps by pressing impregnated long wood against a defibering disc, for example a grinding disc. The wood had been

chopped before the impregnation in order to facilitate the same.

The present invention has the object at this method and at other methods of manufacturing papermaking pulp from lignocellulose material in the form of pieces with a length in the fibre direction of at least 100 mm to achieve an impregnation which is better than at methods used heretofore. This and other objects are achieved by the method according to the invention as it is defined in the attached claims.

The invention is described in greater detail in the following, with reference to the accompanying drawings, in which Fig. 1 shows the moisture quotient of beech wood after compression with different pressures, Fig. 2 shows the density of beech wood after compression with different pressures and after expansion, Figs. 3 and 4 shows an embodiment of a device for carrying out the method according to the invention, Fig. 3 being a vertical section and Fig. 4 a perspective sketch.

The most effective and rapid way of impregnating the wood was found to compress the wood so that the pores are compressed,and then cause the wood to expand below the liquid level. When the pores then are restored, the liquid is sucked into them.

A number of experiments have been carried out to compress beech wood. Blocks of the size 100x100x50 mm were heated to 100°C and thereafter compressed in a laboratory press. The following results were obtained, based on 100 g bone-dry wood.

Pressure MPa	Vol. ml	Dry density in compressed state	Moisture quot. %	Dry solids content %	Vol. after expansion ml	Dry density after expansion kg/dm^3
0	185,5	0,539	73,7	57,6	185,5	0,539
5	174,4	0,573	71,1	58,4	185,5	0,539
7,5	138,8	0,720	61,4	62,0	182,4	0,548
10	118,4	0,844	51,4	66,1	180,4	0,554
12,5	109,5	0,913	45,4	68,8	177,8	0,562
15	103,1	0,970	40,2	71,3	174,6	0,573
17,5	98,2	1,018	38,4	72,3	172,2	0,581
20	97,9	1,021	36,2	73,4	167,2	0,598

The moisture quotient and density after compression are also shown graphically in the diagrams in Figs. 1 and 2. They seem to asymptotically approach values, which well agree with those stated by Kollmann, see above. This implies, that at sufficiently high compression lumen and other cavities are completely compressed, while the submicroscopic pores, which still contain the capillary bound water, remain.

The diagram in Fig. 2 also shows, that the curve for density is very flat at the beginning, proceeds steep in the range of about 5-15 MPa and then deflects to the asymptote.

The density after expansion to zero pressure also has been included in the diagram. This shows, that the greater part of the compression springs back, but for the higher pressure there remains a not insignificant deformation.

The conclusion from these experiments is, that pressures above about 5 MPa must be used for obtaining an effective compression of the wood. A pressure of above

10, preferably 12,5-15 MPa or still higher, 15-30 MPa is desirable. Extrapolation of the curves in Figs. 1 and 2, however, shows that pressures above about 25 MPa scarcely are motivated.

It also is worth to note, that so much liquid has been pressed out, that a dry solids content for the wood of more than 70% was achieved.

According to the embodiment shown in Figs. 3 and 4, the material pieces or staves 1 have a length in the fibre direction of at least 100 mm, suitably at least 200 mm and preferably at least 500 mm and a smallest measure across the fibre direction of at maximum 50 mm, preferably at maximum 25 mm. All staves 1, however, should be of equal length. They have first been softened, for example by steaming.

The staves 1 are directed via a feed shaft 2 into the nip between two counter-rotating rolls 3. The fibre direction of the material is in parallel with the roll axles, and the rolls are slightly longer than the staves. The rolls are mounted in a trough 5 and rotated each by a motor (not shown). The trough is filled with impregnation liquid up to the nip. The nip is adjusted by taking up one roll against the other by means of hydraulic cylinders 4. The rolls can be fluted in order to facilitate the staves to be taken along.

Upon their arrival in the nip the staves are compressed so strongly, that the pores are compressed substantially entirely. Air, water and extractive matter are pressed out thereby. The staves at the same time are rolled out perpendicularly to the fibre direction and thereby assume a greater width, become thinner and get a larger surface. After the nip the material expands again, whereby the pores are restored. The impregnation liquid then is sucked into the pores. The

staves then are transported upward out of the bath by a conveyor 6 to be further processed, for example by digestion in steam phase. The staves thereby are maintained, their fibres oriented in one and the same direction. Thereafter the material is disintegrated to fibre bundles and/or individual fibres. This is achieved preferably by disintegration in a mechanical way to fibres and fibre fragments, for example by pressing the material with liquid added thereto against a rotating defibering member, such as a grindstone, with the fibre direction substantially in the same plane as the defibering surface, against which the material is pressed, and perpendicularly to the direction of movement of said surface.

The digestion or chemical delignification of the impregnated material possibly can be carried out to such an extent, that only insignificant or no mechanical aftertreatment at all is required for exposing the fibres.

For the impregnation different liquids can be used, for example water. Normally an aqueous solution is used, for example of a base, such as caustic soda, a per compound, such as hydroperoxide, a salt of sulphurous acid, such as sodium sulphite or sodium hydrogen sulphite, or a mixture of some of these chemicals.

As an additional effect of the high-pressure pressing the material is deformed, whereby the fibre bond is broken up. This renders it easier for the liquid to penetrate into the substance. Owing to the combined chemical and mechanical action, the fibres partially are set free, so that the continued fibre exposure is facilitated, which spares the fibres and reduces the energy consumption.

A number of experiments have been carried out, partly with aspen wood and partly with fir wood.

Experiments with aspen wood

The aspen wood was dried to a dry solids content of 83%, which corresponds to a moisture quotient of 20%. The wood was sawn to pieces about 100 mm long in the fibre direction and 40x10 mm across the fibre direction. The wood pieces were ground to pulp in a laboratory grinder, partly untreated, partly after impregnation with water, partly after impregnation with a caustic soda solution. The following data applied to the impregnation:

Softening in boiling water, min	0	20	20
Pressure at wood compression, MPa	20	25	25
Liquor concentration, NaOH g/l	0	25	25
Liquid temperature ^{o}C	50	50	50
Reaction time min	0	30	60
NaOH amount taken up in bone-dry wood %	0	2,3	4,7
Acidity of impregnated wood after neutralization with sodium bisulphite pH	-	5,9	6,0

The ground wood was formed to sheets and tested. For comparison, also data for a typical refined chemi-mechanical pulp (CTMP) have been included. The following results were obtained

Pretreatment	None	Water impregnation	NaOH impregn. 2,3%	NaOH impregn. 4,7%	CTMP
Freeness ml CSF	103	272	124	76	100
Beating degree °SR	64	29,5	64,5	74,5	64
Shiver content Somerville %	0,65	3,42	0,72	0,32	-
Fibre fractioning Bauer Mc Nett. %					
+30 mesh	1,6	14,8	4,7	4,6	5
-200 mesh	59,6	29,2	36,6	31,8	25
Density kg/m^3	440	363	449	579	500
Tensile index KNm/kg	8,8	12,7	22,7	39,6	40
Tear index Nm^2/kg	1,06	2,14	2,45	3,06	4
Diffuse blue reflectance factor %	72	73	64	60	55
Diffusion coefficient m^2/kg	79	65	62,5	53	50
Energy consumption KWh/t bone-dry	701	946	930	916	2500

The pulp from the untreated dry wood is very weak, the water-impregnated one is comparable with normal aspen groundwood pulp, while at impregnation with 4,7% NaOH a pulp is obtained, which is comparable with CTMP at refining of chips. The shiver content is very low. The most remarkable feature is that the energy consumption according to the invention was only about 40% of the energy consumption for the CTMP pulp.

Experiments with fir wood

A similar series of experiments was carried out with dried fir wood, which also had a dry solids content of

83%. No softening in boiling water was carried out. The wood was impregnated with a solution of sodium sulphite and then digested in steam phase. The following data applied.

Pressure at wood compression MPa	25	25
Concentration Na_2SO_3 g/l	0	90
Impregnation temperature °C	60	60
Impregnation time min	0	7
Amount of Na_2SO_3 taken up in bone-dry wood %	0	5,0
Digestion temperature °C	-	127
Digestion time min	-	30
Acidity after digestion pH	-	6,5

The following test results were obtained for the ground pulps

Pretreatment	None	Water impregn.	Na_2SO_3	CTMP
Freeness ml CSF	-	40	110	100
Beating degree °SR	72,5	78,5	64	
Shiver content Somerville %	3,4	1,6	2,3	
Fibre fractioning Bauer Mc Nett				
+ 30 mesh	2,9	5,1	26,7	46
-200 mesh	67,8	49,3	29,8	22
Density kg/m^3		477	470	400
Tensile index KNm/kg		26,5	40,2	48
Tear index Nm^2/kg		2,68	4,57	8
Diffuse blue reflectance factor %	62	67	66	61
Diffusion coefficient m^2/kg		71	50	49
Energy consumption kWh/t bone-dry	984	1290	977	2400

As in the case of aspen wood, the pulp of the dry wood was very weak, so that it could not be formed to sheets at all. From the water-impregnated wood a normal groundwood pulp was obtained, while the pulp from the wood, which had been impregnated and digested, is comparable with CTMP. It is, however, slightly cut down, as the fibre fractioning shows. This deteriorates the strength, especially the tearing resistance. The conditions have to be optimized, especially for the grinding.

The energy consumption is only 40% of the energy consumption for CTMP, as in the case of aspen wood.

Other embodiments are possible within the scope of the invention idea. The steaming prior to the compression can be left out or replaced by some other softening. The compression can be carried out in a way other than between rolls, for example between two or more press plates. The impregnation liquid can be added in different ways, for example by spraying on the compressed fibre material.

The invention, of course, is not restricted to the embodiments shown, but can be varied within the scope of the invention idea.

Claims

1. A method of impregnating lignocellulose material in the form of pieces with a length in the fibre direction of at least 100 mm, c h a r a c t e r i z e d i n that the material first is compressed perpendicularly to the fibre direction to such a degree, that its pores are substantially compressed, that the material then is allowed to expand in the impregnation liquid, and that the material thereafter is disintegrated to fibre bundles and/or individual fibres.

2. A method as defined in claim 1, c h a r a c t e r i z e d i n that the pressure at the compression of the material is at least 5 MPa, suitably at least 10 MPa, preferably 15-30 MPa.

3. A method as defined in any one of the preceding claims, c h a r a c t e r i z e d i n that the thickness of the material pieces prior to the impregnation is reduced by cleaving along the fibre direction of the material to a smallest cross-section of at maximum 50 mm, preferably at maximum 25 mm.

4. A method as defined in any one of the preceding claims, c h a r a c t e r i z e d i n that the material pieces are softened prior to the compression.

5. A method as defined in claim 4, c h a r a c t e r i z e d i n that the material pieces are steamed immediately prior to the compression.

6. A method as defined in any one of the preceding claims, c h a r a c t e r i z e d i n that the compression takes place in the nip between two counter-rotating rolls.

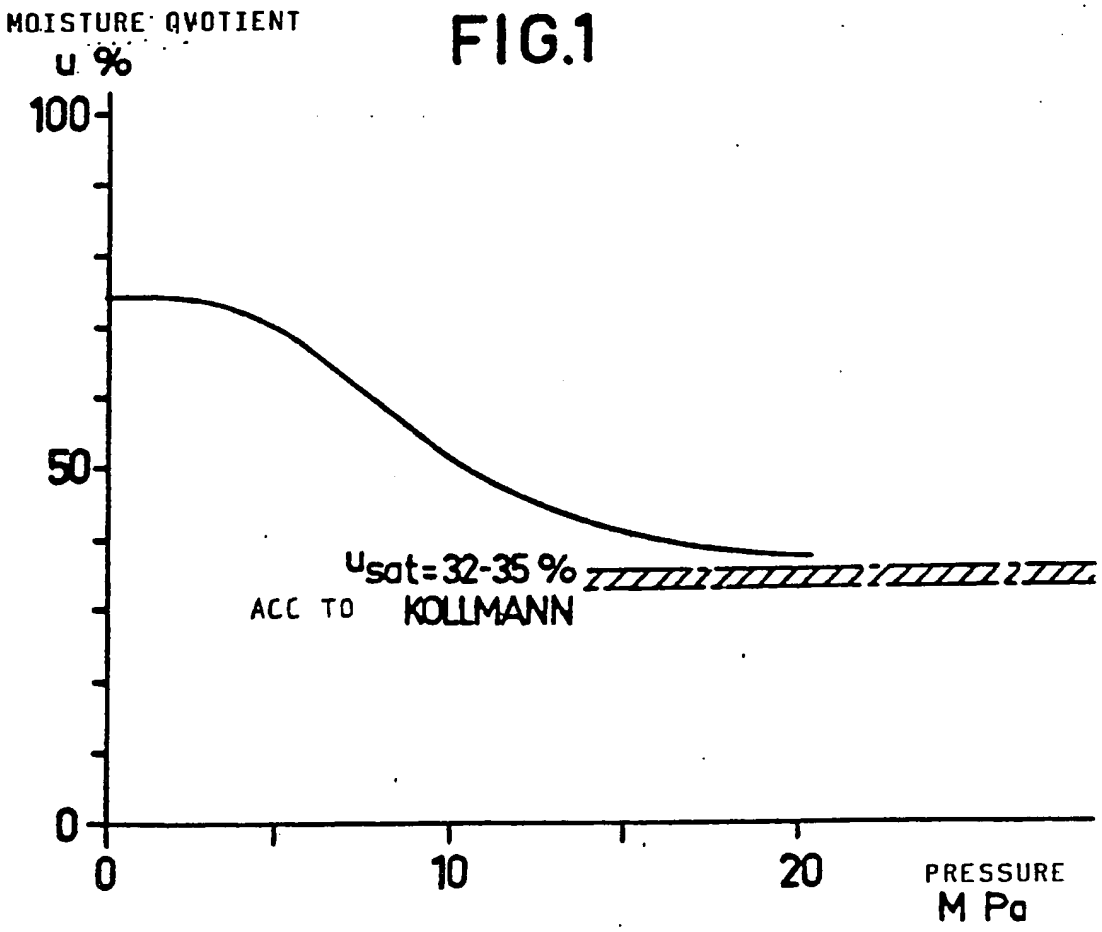
7. A method as defined in claim 6, c h a r a c t e r i z e d i n that the fibre direction of the material is in parallel with the roll axles.

8. A method as defined in any one of the claims 1-5, c h a r a c t e r i z e d i n that the compression takes place between two or more press plates.

9. A method as defined in any one of the preceding claims, c h a r a c t e r i z e d i n that the impregnated material is disintegrated in a mechanical way to individual fibres and fibre fragments.

10. A method as defined in claim 9, c h a r a c t - e r i z e d i n that the impregnated material is disintegrated by pressing it while adding liquid thereto against a rotating defibering member, such as a grindstone, with the fibre direction substantially in the same plane as the defibering surface, against which it is pressed, and perpendicularly to the direction of movement thereof.

FIG.1


MOISTURE QVOTIENT
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0
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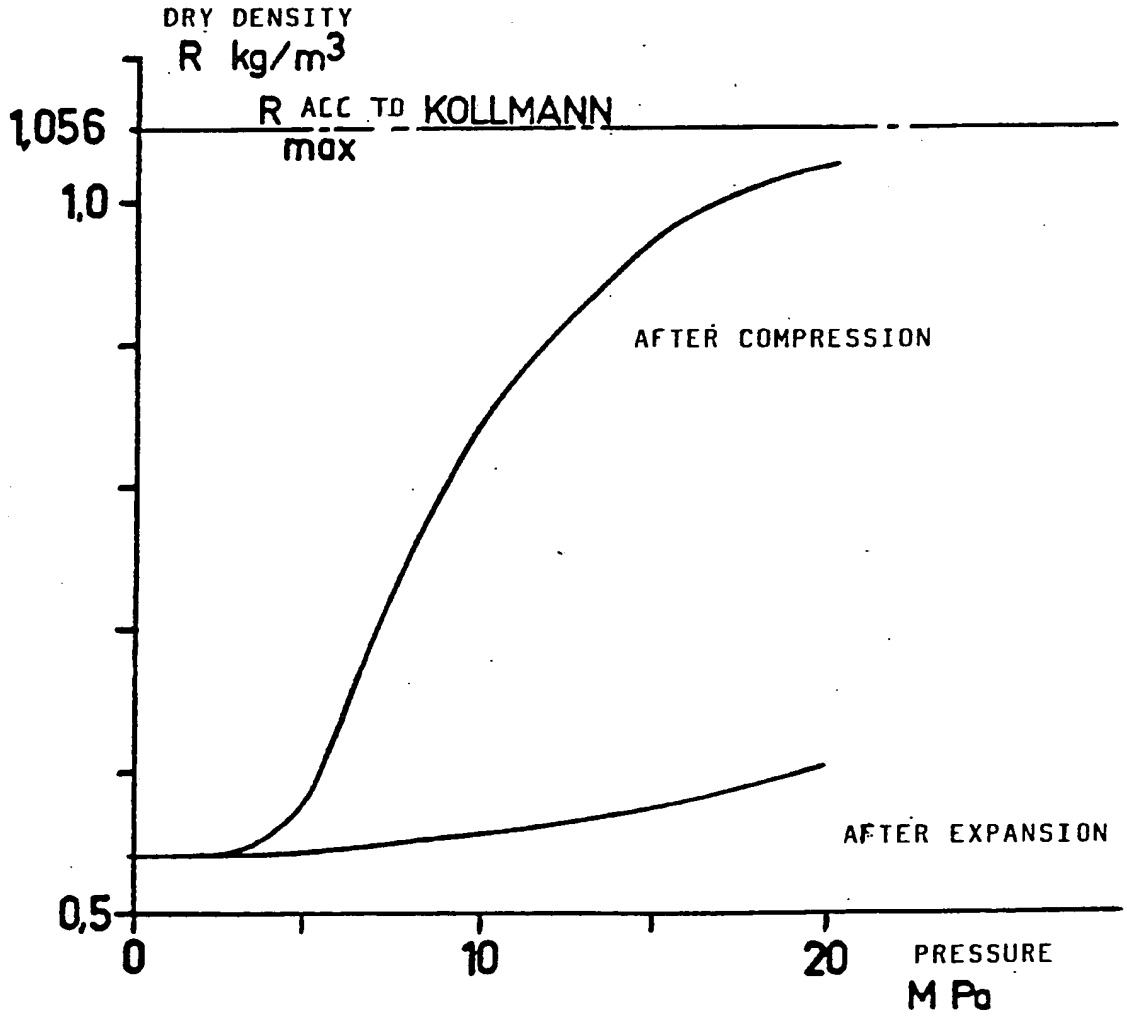
10
20
PRESSURE
M Pa
usat=32-35 %
ACC TO KOLLMANN

FIG.2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AFTER COMPRESSION
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FIG.3

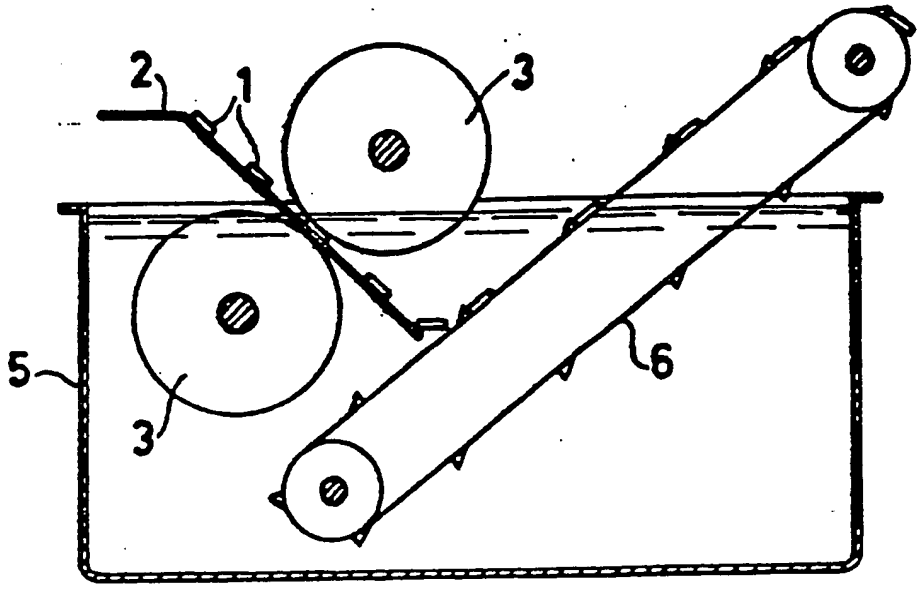
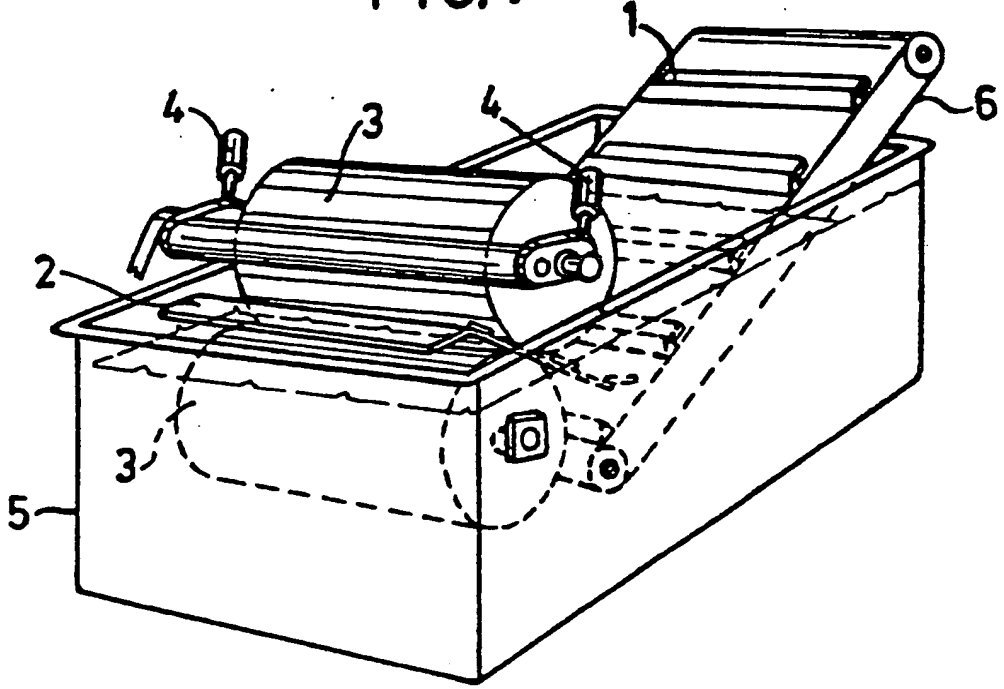


FIG.4



INTERNATIONAL SEARCH REPORT

International Application No PCT/SE88/00458

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC 4

D 21 C 1/10, D 21 B 1/02

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC 4	D 21 B 1/02; D 21 C 1/00-/10
US C1	162: 18, 25, 27, 56

Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched [8]

SE, NO, DK, FI classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category [10]	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
X	TAPPI /May 1979, Vol. 62. No 5, Hardwood hydrogen peroxide chemimechanical pulps, D Lachenal et al. pages 53-57, see spec. page 54	1-10
A	DK, C, 98 799 (A J A ASPLUND) 19 May 1964	
A	US, A, 2 975 096 (M E GINAVEN et al) 14 March 1961 & SE, 227717	

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
1988-12-20	1988 -12- 21
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Swedish Patent Office	Marianne Bratsberg Marianne Bratsberg